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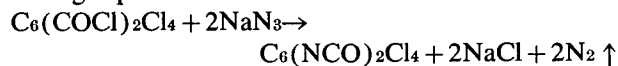
Process Produces Chlorinated Aromatic Isocyanate in High Yield

The problem:

To devise a method of preparing tetrachloro-p-phenylene diisocyanate in high yield.

The solution:

The diisocyanate is prepared by reacting tetrachloroterephthaloyl chloride with sodium azide (in an atmosphere of nitrogen) in accordance with the following equation:



How it's done:

A slurry of sodium azide (1.2 mols) in dry xylene (600 ml) is placed into a three-necked, two-liter flask equipped with a stirrer, thermometer, condenser, dropping funnel, and nitrogen inlet. A solution of tetrachloroterephthaloyl chloride (0.3 mole) in dry xylene (600 ml) is then slowly (over a 45-minute period) mixed into the contents of the flask, maintained under a nitrogen atmosphere. The reaction (with constant stirring of the contents in the flask under nitrogen) is allowed to proceed at 135°C for approximately 15 hours. The resultant slurry is cooled and filtered, and the xylene is evaporated from the filtrate, leaving a residue of approximately 96 percent-pure tetrachloro-p-phenylene diisocyanate in 96.8 percent yield.

Notes:

1. The chlorinated diisocyanate should have application as an intermediate in the preparation of polyurethane foams; the high halogen content would impart added flame resistance to these foams.
2. Inquiries concerning this invention may be directed to:

Technology Utilization Officer
Marshall Space Flight Center
Huntsville, Alabama 35812
Reference: B66-10646

Patent status:

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act [42 U.S.C. 2457 (f)], to the Whittaker Corporation, 3540 Aero Court, San Diego, California 92123.

Source: Floyd Trischler
of the Whittaker Corporation
under contract to
Marshall Space Flight Center

(M-FS-1658)

Category 03